GAS CHROMATOGRAPHY (GC)

• Gas chromatography is a type of chromatography.



- GC is a separation method that exploits the differences in adsorption and partition behaviors of analytes between a mobile phase (He, Nitrogen, etc.) and a stationary phase to separate volatile components in a mixture.
- The instrument that provides the gas chromatographic separation is called the gas chromatograph. A gas chromatograph is a chemical analysis instrument for separating chemicals in a complex sample.

Types of GC

1. Gas-solid chromatography

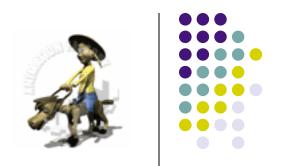


If the stationary phase is a solid, the technique is referred to as gassolid chromatography. The separation mechanism is principally one of adsorption. Today, gas-solid chromatography is not used widely.

2. Gas-liquid chromatography

If the stationary phase is a liquid, the technique is referred to as gasliquid chromatography and the separation mechanisms is principally one of partition. Today, only gas-liquid chromatography is practiced. Therefore, gas-liquid chromatography often just called gas chromatography.

Carrier Gas

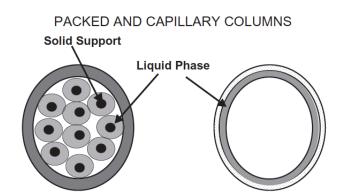


- It is a mobile phase.
- The carrier gas is delivered to the column at a constant flow-rate and pressure.
- Carrier gas does not chemically interact with sample (INERT)
- Typical carrier gases include helium, nitrogen, argon and hydrogen.

Columns



- Columns are responsible for the separation process. Columns, a tube in stainless steel, copper, aluminium or glass, may be shaped like flat, curled or spiral.
- Types of Columns
- 1)Packed columns:
- 2)Capillary columns:



• Packed columns:

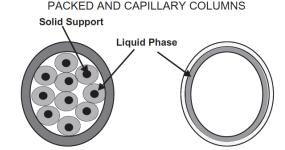
Stationary phase that impregnated an inert solid support is there inside the column.

Most packed columns are 1-3m in length and have an internal diameter of 2-4mm.

• Capillary columns:

Stationary phase is a thin, uniform liquid film coated on the wall of the column. Most capillary columns are 5-60 m in length and have an internal diameter of 0.1-0.53 mm.

"Capillary columns have a more efficient separation of the sample than packed columns."





Types of Capillary Columns



WCOT: Wall Coated Open Tubular

WCOT columns, the wall is directly coated with the stationary-phase layer at a film

SCOT: Support Coated Open Tubular

SCOT columns contain an adsorbed layer of a very fine solid support coated with the liquid phase.

FSOT: Fused Silica Open Tubular

FSOT columns are a new type of WCOT columns

The categories of the stationary phase are:

- Polar stationary phase
- Intermediate polarity stationary phase
- Non-polar stationary phases

Appropriate column choice is governed by to the polarity of components that will be separated.

The solid support that holds the stationary phase should have a large surface area, be chemically inert and have good mechanical strength (Typically diatomaceous earth, glass or organic polymer).



Sample Injection



- The sample inlet should be rapidly.
- The smallest possible sample size should be used.
- A microsyringe is used to inject a liquid sample or gaseous sample (larger volume) through a self-sealing, silicone rubber septum into a flash vaporizer direct injector.
- Split and splitless injections are possible with automatic injector devices in modern GC's

Sampler Injection



- Splitt Injection: In this mode only a small fraction of the 0.2 to 1 µL of sample injected actually enters the separation column.
- Splittless Injection: All of the injected volume is being pushed onto the column.

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The sample analyzed by GC

- must be volatile
- should not be degrade at the temperature of evaporation.

If the sample to be analyzed is non-volatile, derivatives such as trimethylsilyl acyl or ester are prepared and volatilized.

Detectors



- This device measures the quantity of the sample, and it generates an electrical signal. This signal goes to a data system/integrator that generates a chromatogram
- Detectors should be very sensitive.
- Detectors should not be affected by carrier gas flow rate changes.
- The detector temperature should be high enough not to cause condensation of the sample from the column.



Types of GC's Detectors

- Flame Ionisation Dedector (FID)
- Mass Spectrometric Dedector (MS)
- Thermal Conductivity Dedector (TCD)
- Nitrogen-Phosphorus Dedector

*The FID and MS are widely used GC detectors.

Data system/Integrator



According to the detector's signal the data system automatically integrates the peak area, performs calculations.

Factors affectting the separation of substances

- Temperature
- Pressure
- Flow rate of carrier gas
- Column







- The peaks in the chromatogram are characterized by retention time and retention volume.
- Retention time (t_R) is the amount of time a compound spends on the column after it has been injected.
- Retention volume (V_R) is the volume of mobile phase required to elute a solute from the column.

Advantages of GC

- Easily detection and identification,
- Fast analysis (typically minutes),
- Efficient,
- Sensitive,
- Requires small samples (typically μL),
- Reliable,
- Inexpensive.





Disadvantages of GC

- Limited to volatile samples
- Not suitable for thermally labile samples
- Requires spectroscopy (usually mass spectroscopy),

Typical GC Applications

Pharmaceutical

- -raw materials (drug substance)
- -finished products (drug product)
- -Quality control
- -Analysis of essential oils(Rose oil, Thyme Oil)
- Food/Flavors/Fragrances
- Petrochemical
- Chemical/Industrial
- Environmental

